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EP 0 389 235 B1

Description

This invention relates to neutral cure silicone sealants.

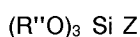
The cure time of a sealant composition based upon a alkoxysilethylene endblocked polymer, alkox-
 5 ysilane cross linker and titanium catalyst is improved by the addition of an oxime compound.

This invention provides a neutral cure silicone composition comprising

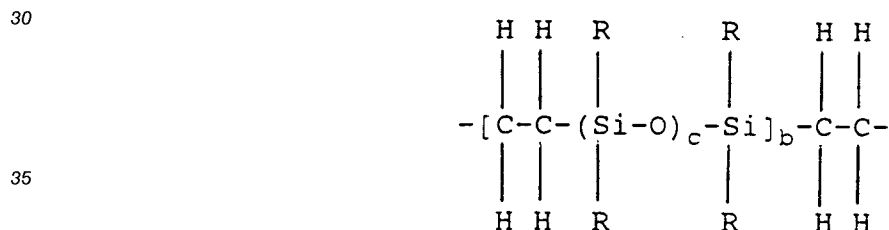
(A) 100 parts by weight of polymer of the formula



where each R is free of aliphatic unsaturation and is selected from the group consisting of monovalent
 20 hydrocarbon, monovalent halohydrocarbon and monovalent cyanoalkyl radicals of 1 to 18 inclusive carbon atoms, each D is selected from the group consisting of vinyl radical and radical of the formula

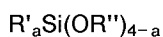


where each R'' is methyl, ethyl, propyl or butyl, Z is a divalent hydrocarbon radical having 2 to 15
 25 carbon atoms or a combination of divalent hydrocarbon radicals and siloxane radicals represented by the formula



where R is as defined above, b is 0 or 1 and c is from 1 to 6; and x is of a value such that the polymer
 40 has a viscosity of from 0.5 to 3000 Pa.s at 25°C, the amount of vinyl radical of D being from 0 to 40 percent of the total of endblocking radicals D,

(B) from 0.1 to 14 parts by weight of a crosslinker of the formula



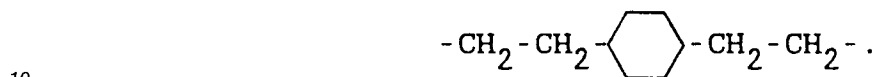
where R' is methyl or phenyl, R'' is methyl, ethyl, propyl or butyl and a is 0, 1 or 2,
 45 (C) from 0.2 to 6.0 parts by weight of titanium catalyst, and

(D) from 0.5 to 4 parts by weight of an oxime compound of the formula

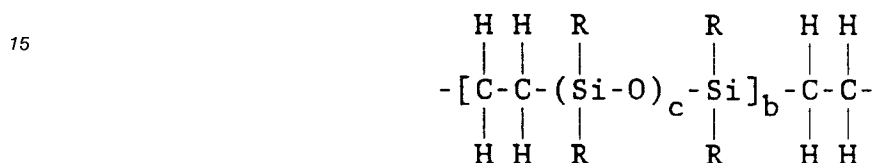


where X is chosen from the group consisting of hydrogen and $\text{R}^5_p \text{Si}$, R^3 is a hydrocarbon of from 1 to 18
 55 carbon atoms, R_4 is a hydrocarbon of from 1 to 18 carbon atoms, R^5 is a hydrocarbon of from 1 to 18 carbon atoms, n is equal to the valence of X and p is 1 or 2.

In the polymer of component (A), R can be any of those monovalent hydrocarbon, monovalent halohydrocarbon or monovalent cyano-alkyl radicals of 1 to 18 inclusive carbon atoms which are known to be useful in silicone sealant materials. The preferred radicals are methyl, ethyl, propyl, phenyl and trifluoropropyl. Z is a divalent hydrocarbon radical or combination of divalent hydrocarbon radicals and siloxane radicals. The divalent hydrocarbon radical can be from 2 to 15 carbon atoms in the form of a
 5 divalent alkylene or arylene radical such as ethylene, propylene, hexylene, phenylene and

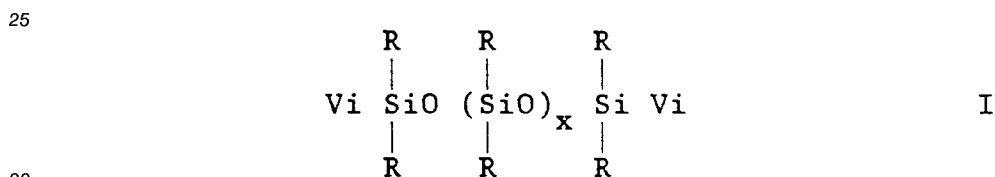


A preferred Z may be represented by the formula

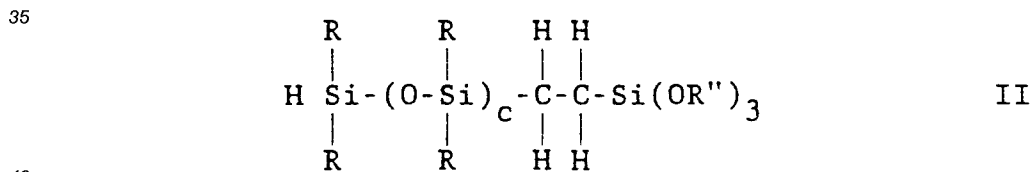


20 where R is as defined above, \underline{b} is 0 or 1 and c is from 1 to 6.

The polymer of (A) can be produced by reacting a vinyl endblocked polydiorganosiloxane of the formula



where each R is as defined above, Vi is vinyl radical and \underline{x} is as defined above with an endcapping composition of the formula



where R and R'' are as defined above and \underline{c} is 1 to 6. This endcapping composition can be produced by a method comprising

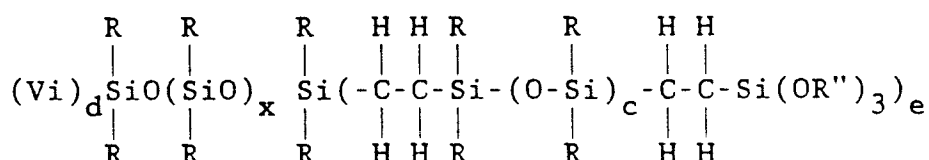
(A) mixing 1 mole of a composition (a) of the formula



50 where R'' is as defined above, with greater than 2 moles of a composition (b) of the formula

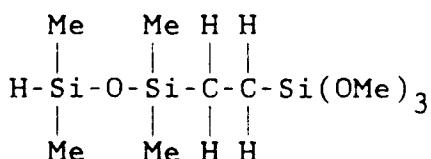


where R and \bar{c} are as defined above, in the presence of a platinum catalyst and allowing to react, then, (B) optionally stripping the excess composition (b) from the product, to give an endcapping composition of the formula as given above. A preferred endcapping composition is that obtained when \bar{c} is equal to 1 and \bar{b} is equal to 1. The above endcapping composition, its method of manufacture and its use in the manufacture of silicone sealants, having an alkoxy functional silane crosslinker and a titanium catalyst, is taught in U.S. Patent 4,772,675, issued September 20, 1988, which shows the endcapping composition, its manufacture and its use. The polymer produced from the above reaction of vinyl endblocked polydiorganosiloxane (I) and endcapping composition (II) can be represented by the formula



where each R, R'', Vi, \bar{c} and \bar{x} , are as defined above and \bar{d} and \bar{e} are chosen so that \bar{d} is on average from 0 to 40 percent of the total of \bar{d} plus \bar{e} .

The amount of the endcapping composition (II) used to react with the vinyl endblocked polydiorganosiloxane (I) is chosen so that the desired number of the vinyl endblocking groups are replaced with the alkoxyethylsilene endblocking group from (II) on a molar basis. As an example, when the endcapping composition (II) is of the formula



where Me is methyl radical and the vinyl endblocked polydiorganosiloxane (I) is a polydimethylsiloxane having a viscosity of about 55 Pa·s at 25°C. then the degree of endblocking versus the amount of endblocker used can be estimated from the following:

Parts by Weight of Endblocker	Percent of Alkoxyethylsilene Endblocks
0.9	100
0.8	89
0.7	78
0.6	67
0.5	55

The above polymers can also be produced by using similar siloxanes and silanes in which the location of the hydrogen atom and the vinyl group which react together are reversed.

A crosslinker (B) of the formula $\text{R}'_a\text{Si}(\text{OR}'')_{4-a}$ where R' is at least one selected from the group consisting of methyl, ethyl, propyl, phenyl and vinyl, R'' is as defined above and a is 0, 1 or 2, is added as a moisture scavenger and as a modulus control agent. These alkoxy silanes and their method of manufacture are well known. The amount of crosslinker is from 0.1 to 14 parts by weight, with from 2 to 8 parts most preferred. It is possible to produce useful sealants without using a crosslinker when the polymer of this invention is present because of the functionality of the polymer itself, but from a practical viewpoint, the crosslinker is useful in that it contributes to the excellent shelf life of the sealant.

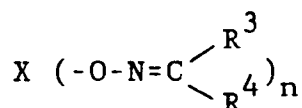
The sealants of this invention are cured through the use of a titanium catalyst (C). The titanium catalyst can be any of those known to be useful in catalyzing the moisture induced reaction of alkoxy containing siloxanes or silanes. Preferred are a titanium catalyst such as titanium naphthenate, titanium esters such as tetrabutyltitanate, tetra-2-ethylhexyltitanate, tetraphenyltitanate, triethanolaminetitanate, organosiloxytitanium compounds such as those described in U.S. Patent No. 3,294,739 and beta-dicarbonyl titanium compounds such as those described in U.S. Patent No. 3,334,067, both patents show titanium catalyst and methods of

manufacture. Preferred catalysts include tetrabutyltitanate, tetraisopropyltitanate, bis-(acetyl-acetonyl)-diisopropyltitanate and 2,5-di-isopropoxy-bisethylacetoacetate titanium. The amount of catalyst is from 0.2 to 6.0 parts by weight per 100 parts by weight of polymer (1). Preferred are from 0.5 to 3.0 parts by weight.

Useful silicone elastomeric sealants are commonly produced with a filler as one of the ingredients.
 5 These fillers are well known in the industry. They are added to the mixture to provide reinforcement of the polymer so as to control the physical properties of the sealant after curing. Reinforcing fillers, such as fumed silica, precipitated silica and diatomaceous earth, are used to give the highest physical strengths to the sealants. Reinforcing fillers are generally recognized as being very fine particles having a surface area from about 50 to 700 m²/g. These fillers may be used with untreated filler surfaces or with treated filler
 10 surfaces, the treatment being used to modify the filler surface so that it properly reacts with the polymer and the other ingredients in the sealant. Calcium carbonate fillers are now available which are produced by precipitation which have a surface area of about 20 m²/g that give a reinforcing effect also. Extending fillers such as titanium dioxide, zirconium silicate, calcium carbonate, iron oxide, ground quartz and carbon black may be used. The amounts of filler used can obviously be varied within wide limits in accordance with the
 15 intended use. For example, in some cases, the sealant could be used with no filler, but it would have very low physical properties. Reinforcing fillers are commonly used in amounts from about 5 to 60 parts by weight to give the highest physical properties, such as tensile strength. Extending fillers are finely ground in that the average particle size is in the range of from about 1 to 10 micrometers. Extending fillers are used to modify the sealant properties and to provide opacity in some cases. Extending fillers are used in
 20 amounts as high as 200 parts by weight and more.

The improved cure rate obtained by the composition of this invention is due to the inclusion in the composition of from 0.5 to 4 parts by weight of an oxime compound of the formula

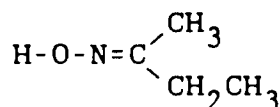
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30 where X is chosen from the group consisting of hydrogen and R⁵_pSi, R³ is a hydrocarbon of from 1 to 18 carbon atoms, R⁴ is a hydrocarbon of from 1 to 18 carbon atoms, R⁵ is a hydrocarbon of from 1 to 18 carbon atoms, n is equal to the valence of X and p is 1 or 2. R³, R⁴ and R⁵ are chosen from radicals such as methyl, ethyl, propyl, phenyl, 3,3,3-trifluoropropyl, isopropyl, octadecyl and benzyl. The preferred radicals are methyl and ethyl, p is preferably 1 and n is 3.

35 A preferred oxime is obtained when X is hydrogen and R³ and R⁴ are methyl or ethyl radicals. When methyl and ethyl are used as R³ and R⁴ respectively a preferred oxime is methylethylketoxime, a commercial product having the formula

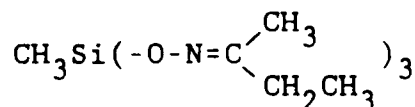
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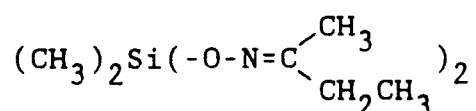
Another preferred oxime compound is methyltrioximosilane of the formula

50



which is obtained through the reaction of methyltrichlorosilane with at least twice the stoichiometric amount of methylethylketoxime.

55 When p in the above formula for the oxime compound is equal to 2, R³ is methyl radical and R⁴ is ethyl radical the oxime compound is of the formula



5

The oxime compound is added in an amount of from 0.5 to 4 parts by weight per 100 parts of polymer (A). The preferred amount of oxime compound is from 0.5 to 1 part per 100 parts of polymer (A).

10 The neutral cure silicone compositions of this invention have a faster cure than the comparable composition made without the presence of the oxime compound. The faster curing compound can be further combined with other ingredients, such as filler, to provide a faster curing sealant. This faster curing sealant is useful in all of the commonly recognized uses of such materials.

15 A preferred method of producing the composition of this invention mixes the polymer (A), with filler, if desired, deairing and adding a deaired mixture of crosslinker (B), titanium catalyst (C) and oxime compound (D), which are added in the absence of exposure to moisture. The crosslinker (B), titanium catalyst (C) and oxime compound (D) can be added separately or they can be mixed together and added as a mixture. The ingredients are stirred to give a uniform mixture. The uniform mixture is then preferably deaired and sealed into storage containers, sealant tubes for example, to store it until it is to be used.

20 The following examples are included for illustrative purposes only and should not be construed as limiting the invention which is properly set forth in the appended claims. Parts are parts by weight.

Example 1

25 A base composition was prepared by mixing 100 parts of a trimethoxysilylethylene endblocked polydimethylsiloxane having a viscosity of about 50 Pa•s at 25°C., 30 parts of a trimethylsilyl endblocked polydimethylsiloxane having a viscosity of about 0.1 Pa•s at 25°C. and 175 parts of a calcium carbonate filler treated with stearate. The base was mixed and then deaired to remove air and moisture from the base.

30 The base (100 parts) was then mixed, in the absence of moisture, with 2 parts of methyltrimethoxysilane cross-linker and 0.52 parts of tetrabutyltitanate to give a composition (1) which cured in the presence of moisture. The composition was stored in a container in the absence of moisture. The cure rate was determined by measuring the tack free time (TFT) of the composition. The tack free time is defined as the time in minutes required for a curing material to form a non-tacky surface film. A sample is spread on a clean smooth surface and timing is begun. Periodically, a clean strip of polyethylene film is laid upon a fresh surface and a one ounce weight applied to it. After 4 seconds, the weight is removed and the strip
35 gently pulled off. The time when the strip pulls cleanly away from the sample is recorded as the tack free time. The result is shown in Table I.

A composition (2) was prepared by repeating the above, except there was also added 0.52 part of methylethylketoxime. This composition was tested for cure rate as above.

40 A composition (3) was prepared by mixing 100 parts of the above base with 2.3 parts of dimethyldimethoxysilane and 0.52 parts of tetrabutyltitanate. The composition was tested for TFT as above, with the result shown in Table I.

A composition (4) was prepared in the same manner as composition (3), except 0.52 part of methylethylketoxime was added. The composition was tested for cure rate as above.

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The cure time of each sample was measured as above, with the results shown in Table II.

Composition	Ketoxime part	Tack Free Time minutes
5*	0.0	56
6*	1.0	>80
7*	0.0	120
8*	1.0	>120
* comparative example		

These comparative examples show that adding the oxime compound to a composition which includes a hydroxyl endblocked polydiorganosiloxane rather than the required vinyl or trialkoxysilethylene endblocked polymer does not result in a faster cure.

Example 3

A series of compositions were prepared in which the polymer used was endblocked with both vinyl groups and trimethoxysilethylene groups.

A polymer was prepared by reacting 100 parts of dimethylvinylsiloxyl endblocked polydimethylsiloxane having a viscosity of about 55 Pa•s at 25 °C. with 0.7 part of endcapper, the endcapper being tetramethyldisiloxane having a trimethoxysilethylene group on one end and a hydrogen atom on the other end. The reaction was run in the presence of 0.01 part of chloroplatinic acid complex of divinyltetramethyldisiloxane diluted with dimethylvinylsiloxyl endblocked polydimethylsiloxane to provide 0.7 weight percent platinum as a catalyst. The reaction mixture was stirred for 2 hours, then allowed to age overnight. The finished polymer was endblocked on average with about 78 percent trimethylsilethylene and 22 percent vinyl. A sealant was then prepared by mixing 100 parts of the above polymer with 8 parts of fumed silica having a surface area of about 150 m²/g, 7 parts of methyltrimethoxysilane and 2 parts of 2,5-di-isopropoxy-bis-ethylacetoacetate titanium, to give comparative sample 9.

Another sample (10) was prepared in a similar manner, but also contained 1 part of methyltri-ox-imosilane (MTO). Each sample was measured for cure rate, with the results as shown in Table III.

Table III	
Composition	Tack Free Time minutes
9*	60
10	33
* comparative example	

Example 4

A composition was prepared in which the polymer was a mixture of the polymer of Example 3 and a hydroxyl endblocked polydimethylsiloxane.

A sealant was prepared by mixing 90 parts of the polymer of example 3, 10 parts of hydroxyl endblocked polydimethylsiloxane fluid having a viscosity of about 50 Pa•s at 25° C. and a hydroxyl content of about 0.057 weight, 8 parts of fumed silica having a surface area of about 150 m²/g, 8 parts of methyltrimethoxysilane and 2 parts of 2,5-di-isopropoxy-bis-ethylacetoacetate titanium, to give comparative sample 11.

Another sample (12) was prepared in a similar manner, but also contained 1 part of methyltri-ox-imosilane (MTO). Each sample was measured for cure rate, with the results as shown in Table IV.

Table IV	
Composition	Tack Free Time minutes
11*	44
12	30
* comparative example	

Example 5

A polymer was prepared as in Example 3, but the amount of endcapper used was 1.1 part rather than 0.7 part. This gave a polymer which was completely endblocked with trimethylsilyl groups.

A sealant was then prepared by mixing 100 parts of the above polymer with 10 parts of fumed silica having a surface area of about 150 m²/g, 10 parts of dimethyldimethoxysilane and 1.6 parts of tetrabutyltitanate, to give comparative sample 13.

Another comparative sample (14) was prepared in a similar manner, but also contained 0.5 part of MTO. Comparative sample (15) was prepared in a similar manner, but contained 1.0 part of MTO. Each sample was measured for cure rate, with the results as shown in Table V. The skin over time is defined as the time required for the material to cure to the point where it no longer adheres to a clean fingertip lightly applied to the surface. The cure conditions are 23 °C. and 50 percent relative humidity.

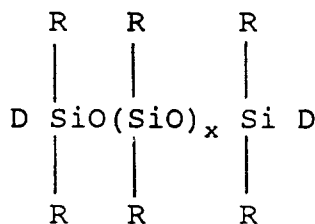
Composition	Ketoxime part	Skin Over Time minutes	Tack Free Time minutes	Slump inches
13*	0.0	12	24	1.13
14*	0.5	6	30	0.13
15*	1.0	8	32	0.05
* comparative example				

The skin over time and the tack free time are much faster than the values as shown in Example 3 because of the use of dimethyldimethoxysilane as the crosslinker rather than methyltrimethoxysilane as in Example 3. The addition of the MTO did not further speed up the cure, probably due to the fact that the cure rate is now controlled by the diffusion rate of moisture into the composition. The amount of MTO present does control the slump of the composition.

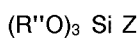
Claims

1. A neutral cure silicone composition comprising

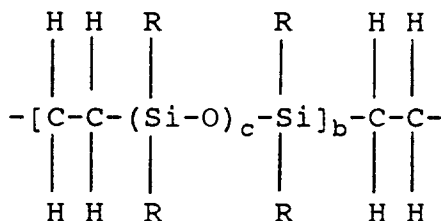
(A) 100 parts by weight of polymer of the formula



where each R is free of aliphatic unsaturation and is selected from the group consisting of monovalent hydrocarbon, monovalent halohydrocarbon and monovalent cyanoalkyl radicals of 1 to 18 inclusive carbon atoms, each D is selected from the group consisting of vinyl radical and radical of the formula

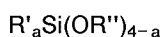


where each R'' is methyl, ethyl, propyl or butyl, Z is a divalent hydrocarbon radical having 2 to 15 carbon atoms or a combination of divalent hydrocarbon radicals and siloxane radicals represented by the formula



where R is as defined above, b is 0 or 1 and c is from 1 to 6; and x is of a value such that the polymer has a viscosity of from 0.5 to 3000 Pa.s at 25°C, the amount of vinyl radical of D being from 0 to 40 percent of the total of endblocking radicals D,

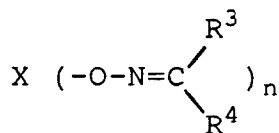
(B) from 0.1 to 14 parts by weight of a crosslinker of the formula



where R' is methyl or phenyl, R'' is methyl, ethyl, propyl or butyl and a is 0, 1 or 2,

(C) from 0.2 to 6.0 parts by weight of titanium catalyst, and

(D) from 0.5 to 4 parts by weight of an oxime compound of the formula



where X is chosen from the group consisting of hydrogen and $R^5_p Si$, R^3 is a hydrocarbon of from 1 to 18 carbon atoms, R^4 is a hydrocarbon of from 1 to 18 carbon atoms, R^5 is a hydrocarbon of from 1 to 18 carbon atoms, n is equal to the valence of X and p is 1 or 2.

2. The composition of Claim 1 wherein (D) is an oxime wherein X is hydrogen and R^3 and R^4 are methyl or ethyl radicals.

3. The composition of Claim 1 wherein (D) is an oximosilane of the formula



4. The composition of Claim 3 wherein R^3 , R^4 and R^5 are methyl or ethyl, p is 1 and n is 3.

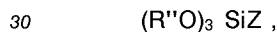
5. The composition of Claim 1 wherein there is also present a filler or fillers.

Patentansprüche

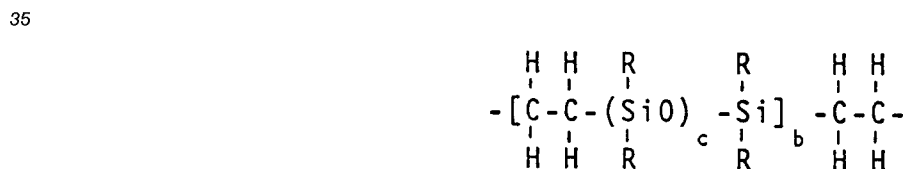
1. Neutral vernetzendes (neutral cure) Silikongemisch, enthaltend
(A) 100 Gewichtsteile eines Polymeren der Formel



in der jeder Rest R frei von aliphatischen Doppel- oder Dreifachbindungen und ausgewählt ist aus der Gruppe, bestehend aus einwertigen Kohlenwasserstoffresten, einwertigen Halogenkohlenwasserstoffresten und einwertigen Cyanalkylresten mit 1 bis einschließlich 18 Kohlenstoffatomen, jeder Rest D ausgewählt ist aus der Gruppe, bestehend aus dem Vinylrest und Resten der Formel



in der jeder Rest R'' Methyl, Ethyl, Propyl oder Butyl bezeichnet, Z einen zweiwertigen Kohlenwasserstoffrest mit 2 bis 15 Kohlenstoffatomen oder eine Kombination aus zweiwertigen Kohlenwasserstoffresten und Siloxanresten der Formel



bedeutet, in der R die zuvor angegebene Bedeutung hat, b einen Wert von 0 oder 1 und c einen Wert von 1 bis 6 besitzt; und in der x einen solchen Wert hat, daß das Polymere eine Viskosität von 0,5 bis 3.000 Pa.s bei 25 °C besitzt, wobei der Anteil der Vinylreste an den gesamten endständigen Resten D 0 bis 40 Prozent beträgt,

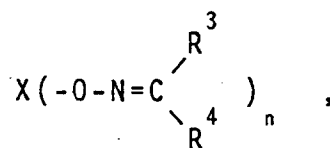
(B) von 0,1 bis 14 Gewichtsteile eines Vernetzungsmittels der Formel



in der R' für Methyl oder Phenyl steht, R'' Methyl, Ethyl, Propyl oder Butyl bedeutet und a für 0, 1 oder 2 steht,

(C) von 0,2 bis 6 Gewichtsteile eines Titankatalysators und

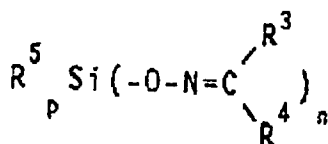
(D) von 0,5 bis 4 Gewichtsteile einer Oximverbindung der Formel



in der X ausgewählt ist aus der Gruppe, bestehend aus Wasserstoff und R^5 Si, R^3 einen Kohlenwasserstoffrest mit 1 bis 18 Kohlenstoffatomen bezeichnet, R^4 einen Kohlenwasserstoffrest mit 1 bis 18 Kohlenstoffatomen bezeichnet, R^5 einen Kohlenwasserstoffrest mit 1 bis 18 Kohlenstoffatomen bezeichnet und n gleich der Valenz von X ist, wobei p für 1 oder 2 steht.

2. Stoffmischung nach Anspruch 1, wobei (D) ein Oxim ist, in dem X Wasserstoff und R^3 sowie R^4 Methyl- oder Ethylreste bedeuten.

3. Stoffmischung nach Anspruch 1, wobei (D) ein Oximsilan der Formel



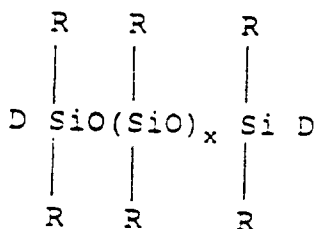
ist.

4. Stoffmischung nach Anspruch 3, wobei R^3 , R^4 und R^5 für Methyl oder Ethyl stehen, p gleich 1 und n gleich 3 ist.

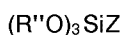
5. Stoffmischung nach Anspruch 1, in der auch ein Füllstoff oder Füllstoffe anwesend ist(sind).

Revendications

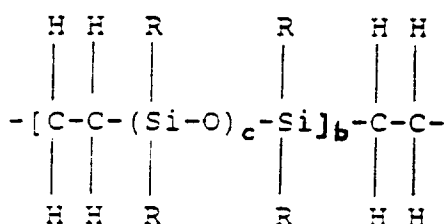
1. Composition de silicone à durcissement neutre, comprenant
(A) 100 parties en poids d'un polymère de la formule



où chaque R est exempt d'insaturation aliphatique et est choisi dans le groupe constitué des radicaux hydrocarbonés monovalents, hydrocarbonés halogénés monovalents et cyanoalkyles monovalents de 1 à 18 atomes de carbone inclusivement, chaque D est choisi dans le groupe constitué du radical vinyle et des radicaux de la formule



où chaque R'' est un groupe méthyle, éthyle, propyle ou butyle, Z est un radical hydrocarboné divalent ayant 2 à 15 atomes de carbone ou une combinaison de radicaux hydrocarbonés divalents et de radicaux siloxanes représentés par la formule



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10 où R est tel que défini ci-dessus, b est 0 ou 1 et c est de 1 à 6 ; et x est d'une valeur telle que le polymère ait une viscosité de 0,5 à 3000 Pa.s à 25 °C, la quantité de radicaux vinyle de D étant de 0 à 40 % de la totalité des radicaux D bloquant les extrémités.

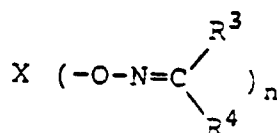
(B) de 0,1 à 14 parties en poids d'un agent de réticulation de la formule

15 $\text{R}'_a\text{Si}(\text{OR}'')_{4-a}$

où R' est un groupe méthyle ou phényle, R'' est un groupe méthyle, éthyle, propyle ou butyle et a est 0, 1 ou 2,

(C) de 0,2 à 6 parties en poids de catalyseur au titane, et

20 (D) de 0,5 à 4 parties en poids d'un composé oxime de la formule



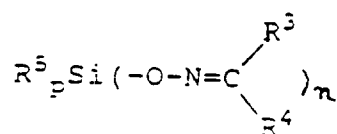
25

où X est choisi dans le groupe constitué de l'hydrogène et de R^5_pSi , R^3 est un hydrocarbure de 1 à 18 atomes de carbone, R^4 est un hydrocarbure de 1 à 18 atomes de carbone, R^5 est un hydrocarbure de 1 à 18 atomes de carbone, n est égal à la valence de X et p est 1 ou 2.

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2. Composition selon la revendication 1, dans laquelle (D) est un oxime où X est de l'hydrogène et R^3 et R^4 sont des radicaux méthyle ou éthyle.

35 3. Composition selon la revendication 1, dans laquelle (D) est un oximosilane de la formule



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4. Composition selon la revendication 3, dans laquelle R^3 , R^4 et R^5 sont des radicaux méthyle ou éthyle, p est 1 et n est 3.

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5. Composition selon la revendication 1, dans laquelle est aussi présente une charge ou des charges.

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